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- [33] **Japan**
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[56]

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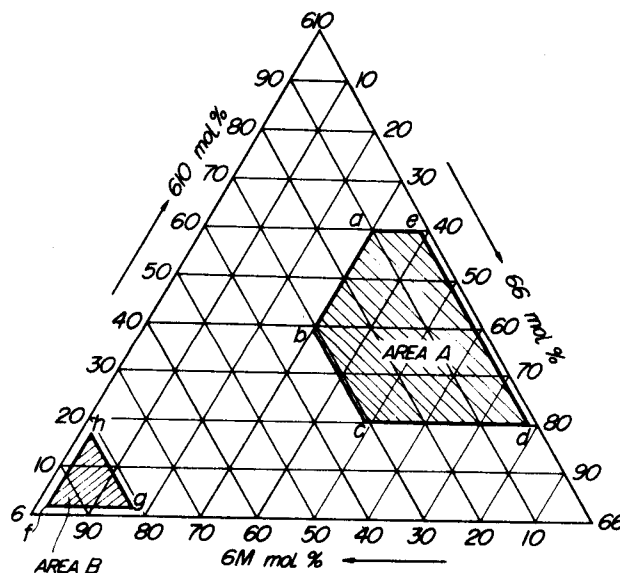
- [54] **CRIMPED COMPOSITE FILAMENTS OF**
POLYCAPROLACTAM OR
POLYHEXAMETHYLENE ADIPAMIDE, AND A
TERPOLYAMIDE OF E-CAPROLACTAM,
HEXAMETHYLENEDIAMMONIUM ADIPATE,
HEXAMETHYLENE DIAMMONIUM SEBACATE
4 Claims, 1 Drawing Fig.

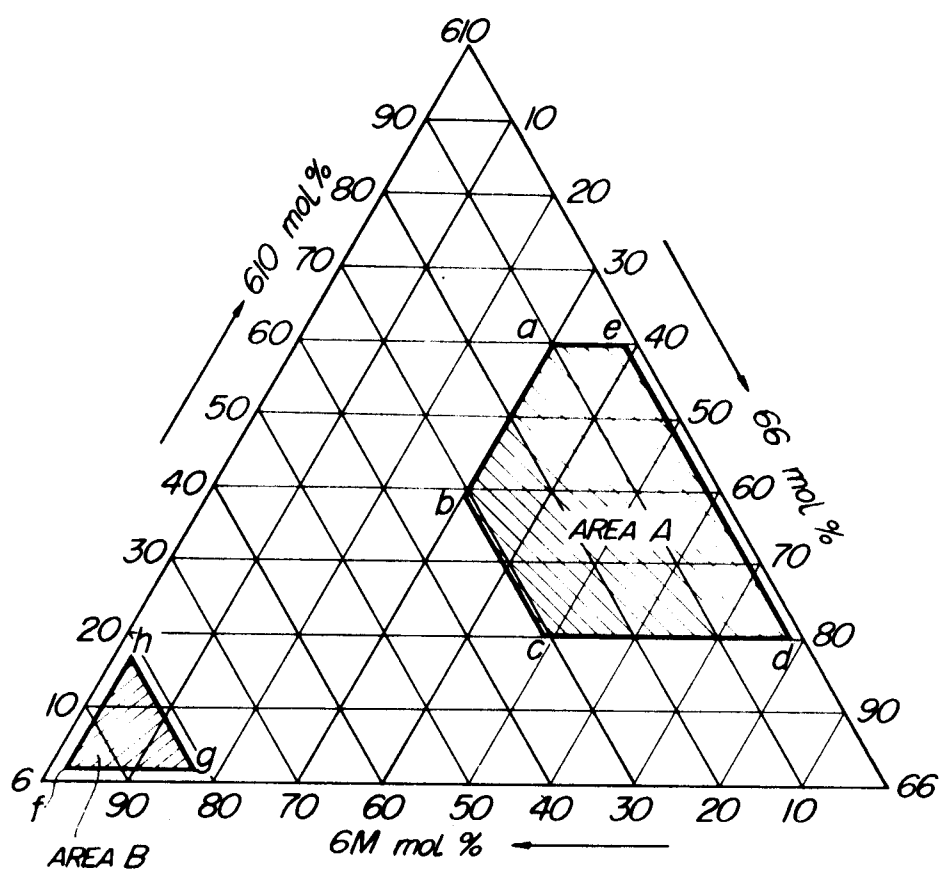
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[51] Int. Cl. D02g 3/00

ABSTRACT: Composite filaments capable of giving excellent crimped yarns are obtained by using, in the production of polyamide composite filaments, the terpolyamide 6/66/610 as a copolymer to be used in combination with either nylon 6 or nylon 66. The proportions of the individual monomers in the terpolyamide are specified in the areas A and B in the accompanying drawing.





**CRIMPED COMPOSITE FILAMENTS OF
POLYCAPROLACTAM OR POLYHEXAMETHYLENE
ADIPAMIDE, AND A TERPOLYAMIDE OF E-
CAPROLACTAM, HEXAMETHYLENEDIAMMONIUM
ADIPATE, HEXAMETHYLENE DIAMMONIUM
SEBACATE**

This invention relates to polyamide-type crimpable composite filaments and a process for their production.

There have been known composite filaments which are produced by separately preparing two spinning solutions of polymers of a different kind, extruding the two spinning solutions through one spinneret, without mixing the solutions to coagulate the solutions and then subjecting the resulting filaments to stretching. These filaments can develop crimps by heat treatment immediately after stretching or even after a certain period of time has elapsed after stretching. Concerning polyamide type composite filaments, a variety of processes have been proposed. However, there have not been obtained composite filaments which are sufficiently satisfactory not only in the crimping force of the filaments but also in such properties as elasticity and bulkiness of crimped yarns obtained therefrom.

The object of the present invention is to provide composite filaments capable of giving crimped yarns which have the above-mentioned properties in combination and are comparable to textured yarns produced according to the conventional false twist method and the like.

The above object can be achieved by the melt spinning process of the present invention in which composite filaments are obtained by using as one polymer, nylon 6 or nylon 66 and as the other polymer a terpolyamide (6/66/610) comprising ϵ -caprolactam (6M), hexamethylenediammonium adipate (66 salt) and hexamethylenediammonium sebacate (610 salt), the feed molar ratios of the components of said polyamide (6/66/610) being limited within the ranges shown, in the accompanying drawing, by the oblique lined areas surrounded by lines formed by connecting the points *a*, *b*, *c*, *d*, *e* and *a* and the points *f*, *g*, *h* and *f*. The drawing is a triangular coordinate showing the feed molar ratios of the individual monomers constituting the terpolyamide to be used in the present invention. In the drawing, the points *a*, *b*, *c*, *d* and *e* of the area A are the points at which the molar ratios of 6M:66 salt : 610 salt are 10:30:60, 30:30:40, 30:50:20, 1:79:20 and 1:39:60, respectively, and the points *f*, *g* and *h* of the area B are points at which said molar ratios are 98 : 1 : 1, 85 : 14 : 1 and 85 : 1 : 14, respectively. The melting point of a terpolymer having a composition within said area A is about 180°–230° C., and that of a terpolymer having a composition within said area B is about 180°–215° C.

If the amounts of 66 salt and 610 salt are outside the proportions in said areas A and B, the terpolymer has a lowered melting point and thus the resulting composite filaments are undesirably deteriorated in thermal resistance. On the other hand, if the amount of 66 salt or 610 salt is excessively small, the composite filaments obtained develop no sufficient crimps.

Even when either nylon 6 or nylon 66 is used as the other component to be conjugate spun, crimps of some degree may be obtained. In view of the easiness of spinning, however, it is advantageous to conjugate spin, in combination, polymers having similar melting point.

The terpolyamide of the present invention is obtained in such a manner that the given amounts of 6M, 66 salt and 610 salt are mixed together and dissolved in water, and the solution is charged in an autoclave and is heated after the atmosphere is substituted by nitrogen, under pressure at 200°–220° C. for several hours. The temperature of the solution is elevated to 250°–300° C., while gradually releasing the pressure, and same is heated at said temperature for 15–20 hours in a nitrogen atmosphere. The other component, i.e. nylon 6 or nylon 66 is obtained by polymerization according to a conventional process. Both components may be incorporated with known stabilizers, pigments and the like.

The spinning in the present invention is effected by the use of a spinneret for conjugate spinning. The stretching may be carried out either at room temperature or at an elevated temperature of 50°–180° C. using a hot plate or the like. In this case, there is a tendency for the crimps to become coarser and for the bulkiness to become more favorable with increasing stretching temperature. The type of arrangement of components may be either side-by-side-type or eccentric sheath-core-type.

The present invention will be illustrated below with reference to the following examples. In the examples, the term "relative" viscosity η rel. of the polymer represents the ratio between the viscosity of a solution formed by dissolving 1 g. of polymer in 100 ml. of 95.5 percent sulfuric acid and the viscosity of the solvent. The term "number of crimps" represents the number of crimps formed, within a length of 25 mm., on a crimped filament when a load of 1 mg./d. has been applied thereto, in which the distance between a peak and an adjacent peak or a bottom to an adjacent bottom of crimps on the crimped filament is counted as one crimp. The "degree of crimp" is defined by the following equation:

$$\text{Crimp elongation} = \frac{l_2 - l_1}{l_1} \times 100\%$$

wherein l_1 is the length of a crimped filament when an initial load of 1 mg./d. has been applied thereto, and l_2 is the length of the filament when the initial load has been removed and a load of 100 mg./d. has been applied in place thereof. The "crimping force" is the number of crimps developed when a straight filament after stretching has been treated under a load of 2 mg./d. in saturated steam at 100° C. for 15 minutes.

EXAMPLE 1

A mixture comprising 800 g. of nylon ϵ -caprolactam, 100 g. of 66 salt and 100 g. of 610 salt (molar ratio of 6 : 66 : 610=91.3 : 4.9 : 3.8) was charged with 200 ml. of water and was fed to an autoclave. After substituting the atmosphere with nitrogen, the mixture was heated under pressure at 220° C. for 2 hours. Thereafter, the temperature was elevated while gradually releasing the pressure. After the temperature had reached 280° C., the pressure was further released to atmospheric pressure, and the mixture was heated at said temperature for 20 hours while flowing nitrogen into the autoclave. Subsequently, the resultant material was extruded under pressure from the autoclave, was shaped into the form of chips by means of a cutter, and was then subjected to hot water extraction to obtain a terpolyamide having a relative viscosity η rel. of 2.8 and a melting point of 185° C.

The thus-obtained terpolyamide and nylon 6 having η rel. of 2.3 and containing 0.3 percent of titanium oxide were extruded in equal amounts through a known side-by-side-type conjugate spinning spinneret having 10 orifices of 0.3 mm. in diameter and were taken up at a rate of 800 m./min. The resulting unstretched filaments were stretched to 3.5 times at room temperature to obtain composite stretched filaments of 50 deniers/10 filaments. The stretched filaments were then subjected in a relaxed state to boiling water treatment at 100° C. to obtain crimped yarns high in elasticity. The crimp properties of the crimped yarns were as follows:

Number of crimps: 84/ 25 mm.
Crimp elongation: 196 percent
Crimping force: 18

EXAMPLE 2

The same unstretched filaments as in example 1 were stretched to 3.5 times on a hot plate at 100° C. and were subjected to the same heat treatment as in example 1 to obtain crimped yarns high in bulkiness and excellent in hand. The crimp properties of the crimped yarns were as follows:

Number of crimps: 46/ 25 mm.
Crimp elongation: 168 percent
Crimping force: 15

EXAMPLE 3

A mixture comprising 850 g. of 6M, 100 g. of nylon 66 salt and 50 g. of nylon 610 salt (molar ratio of 6 : 66 : 610=93.4 : 4.7 : 1.9) was treated in the same manner as in example 1 to obtain a terpolyamide having η rel. of 2.5 and a melting point of 195° C. This terpolyamide, and nylon 6 having η rel. of 2.3 and containing 0.3 percent of titanium oxide were extruded through a known side-by-side-type conjugate spinning spinneret having 30 orifices of 0.3 mm. in diameter and were taken up at a rate of 800 m./min. The resulting unstretched filaments were stretched to 3.0 times on a hot plate at 100° C. to obtain composite stretched filaments of 140 deniers/30 filaments. The stretched filaments were then subjected in a relaxed state to boiling water treatment at 100° C. to obtain

crimped yarns having the following crimp properties:

Number of crimps: 52/ 25 mm.
Crimp elongation: 132 percent
Crimping force: 16

EXAMPLE 4

A mixture comprising 850 g. of 6M, 75 g. of nylon 66 salt and 75 g. of nylon 610 salt (molar ratio of 6 : 66 : 610=93.5 : 3.5 : 3.0) was treated in the same manner as in example 1 to obtain a terpolyamide having η rel. of 2.5 and a melting point of 195° C. This terpolyamide, and nylon 66 having η rel. of 2.4 and containing 0.3 percent of titanium oxide were extruded through a known side-by-side-type conjugate spinning spinneret having 10 orifices of 0.3 mm. in diameter and were taken up at a rate of 800 m./min. The resulting filaments were stretched to 3.0 times on a hot plate at 100° C. to obtain composite stretched filaments of 60 deniers/10 filaments. The stretched filaments were subjected to the crimping treatment according to example 1 to obtain crimped yarns which were excellent in hand and had the following crimp properties:

Number of crimps: 42/ 25 mm.
Crimp elongation: 169 percent
Crimping force: 15

EXAMPLE 5

A mixture comprising 50 g. of 6M, 375 g. of nylon 66 salt and 57 g. of nylon 610 salt (molar ratio of 6 : 66 : 610=11.9 : 39.1 : 49.0) was charged with 100 ml. of water and was fed to an autoclave. After substituting the atmosphere with nitrogen, the mixture was heated under pressure at 220° C. for 2 hours. Thereafter, the temperature was gradually elevated while releasing the pressure, after the temperature had reached 280° C., the pressure was further released to atmospheric pressure, and the mixture was heated at said temperature for 20 hours while flowing nitrogen into the autoclave. Subsequently, the resultant was extruded under pressure from the autoclave, was shaped into the form of chips and was then subjected to hot water extraction to obtain a terpolyamide having η rel. of 2.5 and a melting point of 196° C.

The thus-obtained terpolyamide, and nylon 6 having η rel. of 2.3 and containing 0.3 percent of titanium dioxide were extruded in equal volumes through a known side-by-side-type conjugate spinning spinneret having 10 orifices of 0.3 mm. in diameter and were taken up at a rate of 800 m./min. The resulting unstretched filaments were stretched to 3.5 times at room temperature to obtain composite stretched filaments of 50 deniers/10 filaments. The stretched filaments were subjected in a relaxed state to boiling water treatment at 100° C. to obtain crimped yarns high in elasticity and strong in tenacity. The crimp properties of the yarns were as follows:

Number of crimps: 76/ 25 mm.
Crimp elongation: 296 percent
Crimping force: 22

EXAMPLE 6

The same unstretched filaments as in example 5 were stretched to 3.5 times on a hot plate at 100° C. and were subjected to the same heat treatment as in example 5 to obtain

crimped yarns high in bulkiness. The crimp properties of the yarns were as follows:

Number of crimps: 37/ 25 mm.
Crimp elongation: 118 percent
Crimping force: 15

EXAMPLE 7

A mixture comprising 60 g. of 68-caprolactam, 470 g. of nylon 66 salt and 470 g. of nylon 610 salt (molar ratio of 6 : 66 : 610=14.0 : 47.2 : 38.8) was treated in the same manner as in example 5 to obtain a terpolyamide having η rel. of 2.6 and a melting point of 190° C. This terpolyamide, and nylon 6 having η rel. of 2.3 and containing 0.3 percent of titanium dioxide were extruded through a known side-by-side-type conjugate spinning spinneret having 30 orifices of 0.3 mm. in diameter and were taken up at a rate of 600 m./min. The resulting unstretched filaments were stretched to 3.0 times to obtain composite stretched filaments of 140 deniers/30 filaments. The filaments were then subjected in a relaxed state to boiling water treatment at 100° C. to obtain crimped yarns having the following properties:

Number of crimps: 60/ 25 mm.
Crimp elongation: 190 percent
Crimping force: 17

EXAMPLE 8

A mixture comprising 98.7 g. of 6M, 342 g. of nylon 66 salt and 261 g. of nylon 610 salt (molar ratio of 6 : 66 : 610=29.1 : 43.5 : 27.4) was treated in the same manner as in example 5 to obtain a terpolyamide having η rel. of 2.3 and a melting point of 205° C. This terpolyamide was used as a core component, and nylon 66 having η rel. of 2.3 and containing 0.3 percent of titanium dioxide was used as a sheath component. The two components were extruded, in a copolymer to nylon 66 ratio of 1 : 2, through a known conjugate spinning spinneret and were taken up at a rate of 800 m./min. to obtain so-called kidney-type composite unstretched filaments. The filaments were stretched to 3.5 times on a hot plate at 100° C. to form stretched filaments of 70 deniers/18 filaments. The stretched filaments were then subjected to boiling water treatment at 100° C. to obtain crimped yarns high in bulkiness

The crimp properties of the crimped yarns were as follows:

Number of crimps: 25/25 mm.
Crimp elongation: 98 percent
Crimping force: 10
We claim:

1. A process for producing a side-by-side or eccentric sheath-core crimped composite filaments by melt spinning poly- ϵ -caprolactam or polyhexamethylene adipamide and, a terpolyamide comprising ϵ -caprolactam, hexamethylenediammonium adipate and hexamethylenediammonium sebacate wherein the molar composition of said terpolyamide is within the range defined by the area A in the drawing.

2. A process for producing a side-by-side or eccentric sheath-core crimped composite filaments by melt spinning poly- ϵ -caprolactam or polyhexamethylene adipamide and, a terpolyamide comprising ϵ -caprolactam, hexamethylenediammonium adipate and hexamethylenediammonium sebacate wherein the molar composition of said terpolyamide is within the range defined by the area B in the drawing.

3. A side-by-side or eccentric sheath-core crimped composite filament, one component of said filament being caprolactam, or polyhexamethylene adipamide, the other component being a terpolyamide of ϵ -caprolactam, hexamethylene diammonium adipate, and hexamethylene diammonium sebacate, wherein the ϵ -caprolactam, hexamethylenediammonium adipate and hexamethylenediammonium sebacate are present in a molar ratio of 1-30: 30-79: 20-60, with the proviso that the total amount of the three above components equals 100 percent.

4. A side-by-side or eccentric sheath-core crimped composite filament, one component of said filament being capro-

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lactam, or polyhexamethylene adipamide, the other component being a terpolyamide of ϵ -caprolactam, hexamethylene diammonium adipate, and hexamethylene diammonium sebacate, wherein the ϵ -caprolactam, hex-

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amethylenediammonium adipate and hexamethylenediammonium sebacate are present in a molar ratio of 85-98: 1-14: 1-14, with the proviso that the total amount of the three above components equals 100 percent.

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